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Project Summary

Methods for Level 2 Analysis by Organic Compound Category

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This report presents the results of a laboratory evaluation of specific detailed methods for analysis of organic chemicals in particular compound categories. The report supplements the earlier Level 2 procedures manual[1] and provides an initial experimental data base as a resource for laboratories undertaking Level 2 studies. The Level 2 methods were designed to provide more detailed and more accurate data concerning specific compounds whose presence in the sample could be inferred on the basis of the categorical analysis of Level 1.

A systematic laboratory evaluation of the methods for Level 2 analysis of each major category of organic compounds was conducted. Methods were evaluated using sets of model compounds spanning a range of molecular weights and boiling points in each category. Evaluation criteria included: detection limit, retention time, chromatographic resolution, and precision and accuracy of the methods.

Specific detailed Level 2 methods have been recommended in the full report for 25 categories of organic compounds. Most of the primary recommendations are based on packed column gas chromatography/mass spectrometry, with alternative procedures suggested where appropriate for particular sample matrices or compound types. Analytical data using the specified example conditions are usually illustrated for each recommended method.

The strategy for application of the recommended Level 2 methods to real environmental samples is illustrated by a partial Level 2 analysis of a combustion effluent sample. The Level 1 data on the sample are reviewed to illustrate the Level 1 to Level 2 transition process and decision criteria. The Level 2 analysis provided qualitative confirmation of compound classes, subcategories, and specific compounds identified at Level 1. Level 2 quantitative data agreed with expectations based on Level 1 chromatographable organics. However, comparison of the partial Level 2 data with the total quantity of organics found by Level 1 for this sample illustrates that additional types of analysis methods will need to be developed to make possible complete Level 2 analysis of the range of organics identifiable at Level 1.

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

This report represents a step in the development of a general methodology for chemical and biological assessment of environmental effects of industrial effluents. It is a supplement to the Level 2 procedures manual[1] for sampling and analysis of organics which was

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published in 1979. The procedures manual[1] presented concepts and general guidelines for Level 2 sampling and analysis. It also suggested specific analysis procedures that were considered to be appropriate for particular organic compound categories.

Level 1/Level 2 in the Phased Approach

Both this report and the earlier procedures manual were prepared to support IERL-RTP's phased approach to the overall environmental assessment program. IERL-RTP's Process Measurements Branch has developed a three-phased approach to sampling and analysis for environmental assessment. The philosophy of this approach, which has been described elsewhere[2], is briefly described here to provide a context for the present document.

Level 1, the first phase of sampling and analysis for environmental assessment, is designed to provide enough information about the composition of effluent and process streams to permit them to be ranked in order of priority with regard to probable environmental hazard. The Level 1 assessment is intended to: (1) provide preliminary environmental assessment data; (2) identify principal problem areas; and (3) formulate the data needed for ranking of energy and industrial processes, streams within a process, components within a stream, and classes of materials for further consideration in the overall assessment. The second phase of sampling and analysis effort, Level 2, is designed to provide additional information that will confirm and expand the information gathered in Level 1. The third phase, Level 3, involves quantitative monitoring of specific pollutants to provide information for control device design, development, and evaluation.

The objective of a Level 2 study will be to obtain more detailed and accurate data about the composition of a particular process stream than is available in the context of a Level 1 study. The improved accuracy should be either primarily quantitative in terms of establishing a truly representative emission rate, primarily qualitative in identifying organic species, or both.

Level 2 inquiries are expected to be directed primarily at the identification, quantification, and confirmation of specific compounds whose presence could be inferred on the basis of the categorical analysis of Level 1.

Based on information available to date, it appears that mass spectrometric analysis of sample extracts, using the direct insertion probe, is an attractive approach to obtaining Level 2 information on the non-chromatographable GRAV range materials identified in Level 1 analysis. High resolution mass spectrometry (HRMS) can provide better qualitative information than available from the Level 1 low resolution mass spectrometry (LRMS). The quantitative estimates provided by Level 1 LRMS can be improved by applying the method of known addition to the LRMS (or HRMS) analysis for particular compounds suspected to be present at levels of concern.

If Level 1 analysis shows a category to be present at concentrations that exceed the emission level criteria for a particular environmental assessment, then Level 2 information will generally be needed to determine whether/which particular members of that category are present and to obtain better quantitative data. For most organic compound categories, the specific organic compounds that are known to be of concern (with regard to potential impacts on health and/or the environment) are the lower molecular weight numbers of the category, for which toxicological and ecological effects data exist. Therefore, the most immediate need was to develop and evaluate Level 2 methods that are applicable to this molecular weight range. The analytical methods presented in the report, which were selected primarily to meet that need, therefore rely heavily on gas chromatography/mass spectrometry (GC/MS) as a broad but selective technique for analysis of low to moderate molecular weight organics.

The report presents the results of a laboratory investigation and evaluation of the set of detailed procedures for analysis of organic compounds by category recommended in the February 1979 Level 2 procedures manual. Those recommendations had evolved, based on careful review of literature and discussions with other workers experienced in the chemical analysis of organic pollutants, from a list of possible procedures in the interim Level 2 manual.[1] The laboratory evaluation of the recommended procedures had two objectives:

- To determine whether the original recommendations were appropriate with or without minor modifications.
- To provide appropriate analytical data to users of the Level 2 analytical techniques.

The experimental detail for each method evaluated is provided in the full report. The basic approach was to evaluate each method using a test mixture of model compounds representative of that category. Criteria for method evaluation included: lower limits of analysis, analysis time, chromatographic resolution, precision and accuracy. The result of these experiments is a set of analytical procedures with supporting data available, including specific analytical conditions, chromatographic retention times, lower limits of analysis, and precision and accuracy data. Relevant data from other sources were also included when available.

References

1. Harris, J.C., M.J. Hayes, P.L. Levins, and D.B. Lindsay, "EPA/IERL-RTP Procedures for Level 2 Sampling and Analysis of Organic Materials", EPA-600/7-79-033, February 1979.

   EPA-600/7-79-033, February 1979 and Harris, J.C., and P.L. Levins, "EPA/IERL-RTP Interim Procedures for Level 2 Sampling and Analysis of Organic Materials, EPA-600/7-78-016, February 1978.

2. Dorsey, J., C.H. Lochmuller, L.D. Johnson, and R. Statnick, "Guidelines for Environmental Assessment Sampling and Analysis Programs - Level 1," U.S. Environmental Protection Agency, RTP.

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The complete report, entitled "Methods for Level 2 Analysis by Organic Compound Category," (Order No. PB 81-179 855; Cost: $24.50, subject to change) will be available only from:
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